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Copper-mediated trifluoromethylation of diaryliodonium salts with difluoromethyltriflate

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ABSTRACT

The reaction of diaryliodonium salts with difluoromethyltriflate in the presence of TBAT and CuTC gave the corresponding trifluoromethylated arenes in moderate yields. Compared to other difluorocarbene-derived trifluoromethylation reactions, the current one proceeded at mild reaction conditions (room temperature) within short reaction time (5 min).

1. Introduction

The growing presence of fluorine in pharmaceuticals and agrochemicals [1] has recently stimulated a renewed interest in synthetic methods of fluorinated compounds [2,3]. Especially, tremendous efforts have been devoted to the development of new processes for the direct introduction of the trifluoromethyl group into organic molecules [3]. Over the past several years, the transition-metal-mediated/catalyzed trifluoromethylation of aromatic compounds with electrophilic [4], nucleophilic [5], and radical [6] sources of CF_3 has become a field of intense research effort. Despite great achievements have been made in this research area, with the success of positron emission tomography (PET) and the consequent upsurge of interest in [^{18}F]radiochemistry [7], there is a pressing need for development of new trifluoromethylation methods, which are potentially useful for PET imaging.

Up to now, all of the reported [^{18}F]trifluoromethylation of aromatic compounds relied on the difluorocarbene/fluoride/copper multicomponent strategy [8]. This difluorocarbene-derived trifluoromethylation strategy was independently developed by Burton and Chen in the early 1990s [9]. They found that the reactions of aryl halides and methyl halodifluoroacetate in the presence of potassium fluoride and copper iodide afforded trifluoromethylated products (Scheme 1a). Based on the same strategy, Zhang and co-workers recently developed a new reagent, trimethylsilyl chlorodifluoroacetate (TCDA), for the synthesis of trifluoromethylated (hetero) arenes (Scheme 1b) [10]. Although these reactions are efficient, normally high reaction temperature (90–120 °C) and long reaction times (1–10 h) are required. Thus, the development of the new difluorocarbene-derived trifluoromethylation reactions under mild reaction conditions within short time remains a considerable unmet need. Very recently, we reported a convenient method for the preparation of trifluoromethylated arenes from the copper-mediated coupling reaction of diaryliodonium salts with $TMSCF_3$ at room temperature [11a]. Inspired by this work, we envisioned that diaryliodonium salts should also served as suitable coupling partners in difluorocarbene-derived trifluoromethylation reactions. Owing to the highly electron-deficient nature and excellent leaving-group ability [12], diaryliodonium salts are expected to undergo fast transformations, which should enable the use of much milder reaction conditions compared to the trifluoromethylation of aryl halides. As a part of our ongoing research in the development of new methods for trifluoromethylation reaction [11], we describe here the copper-mediated trifluoromethylation of diaryliodonium salts with difluoromethyltriflate. This protocol is highlighted by the mild reaction conditions (room temperature) and short reaction time (5 min).

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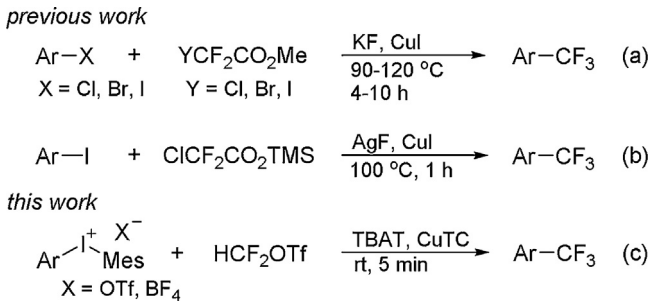
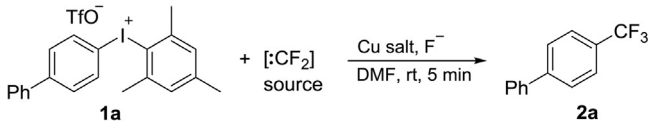


Scheme 1. Difluorocarbene-derived trifluoromethylation of aromatic compounds.

2. Results and discussion

Initially, [1,1′-biphenyl]-4-ylmesityliodonium triflate (**1a**) was chosen as the test substrate to optimize the reaction conditions (Table 1). Treatment of **1a** with $[Ph_3PCF_2Br]Br$, a well-known difluorocarbene precursor [13], in the presence of $CuBF_4{\cdot}(CH_3CN)_4$ and KF at room temperature could not give the desired product **2a** (entry 1). After the addition of 18-crown-6, product **2a** was formed in 8% yield (entry 2). Then, other common difluorocarbene precursors including $ClCF_2CO_2Me$ [14], $TMSCF_2Br$ [15], and HCF_2OTf [16] were tested. To our delight, the yield of **2a** was obtained in 57% yield when HCF_2OTf was used (entry 5), while $ClCF_2CO_2Me$ and $TMSCF_2Br$ were not effective (entries 3 and 4). Although other copper salts, such as CuCl, CuI, CuSCN, $(CuOTf)_2{\cdot}C_6H_6$ and CuTC, could also promote this reaction (entries 6–10), only CuTC shown better reactivity than $CuBF_4{\cdot}(CH_3CN)_4$, affording **2a** in 62% yield (entry 10). Subsequently, different fluoride sources including CsF, TBAF, and TBAT were screened (entries 11–13). Among them, TBAT was proven the optimal one to give the highest yield of **2a** (77%) (entry 13). It was noteworthy that this yield was obtained in only 5 min and no higher yield was observed after longer time. To improve the reaction yield further, the ligand (phen) or base (*t*-BuOK) was added to the reaction mixture (entries 14 and 15). However, the yield of **2a** was not raised.

Table 1
Optimization of reaction conditions.[a]



Entry	[:CF_2] source	Cu salt	F source	Yield (%)[b]
1	$[Ph_3PCF_2Br]Br$	$CuBF_4{\cdot}(CH_3CN)_4$	KF	0
2	$[Ph_3PCF_2Br]Br$	$CuBF_4{\cdot}(CH_3CN)_4$	KF/18-crown-6	8
3	$ClCF_2CO_2Me$	$CuBF_4{\cdot}(CH_3CN)_4$	KF/18-crown-6	0
4	$TMSCF_2Br$	$CuBF_4{\cdot}(CH_3CN)_4$	KF/18-crown-6	0
5	HCF_2OTf	$CuBF_4{\cdot}(CH_3CN)_4$	KF/18-crown-6	57
6	HCF_2OTf	CuCl	KF/18-crown-6	12
7	HCF_2OTf	CuI	KF/18-crown-6	45
8	HCF_2OTf	CuSCN	KF/18-crown-6	15
9	HCF_2OTf	$(CuOTf)_2{\cdot}C_6H_6$	KF/18-crown-6	12
10	HCF_2OTf	CuTC	KF/18-crown-6	62
11	HCF_2OTf	CuTC	CsF	58
12	HCF_2OTf	CuTC	TBAF	trace
13	HCF_2OTf	CuTC	TBAT	77
14[c]	HCF_2OTf	CuTC	TBAT	63
15[d]	HCF_2OTf	CuTC	TBAT	50

[a] Reaction conditions: **1a** (0.1 mmol), Cu salt (0.1 mmol), [:CF_2] source (0.4 mmol), F source (0.3 mmol), DMF (2.0 mL), under N_2, rt, 5 min.
[b] Yields were determined by ^{19}F NMR spectroscopy using trifluoromethoxylbenzene as an internal standard.
[c] Phen (0.1 mmol) was added.
[d] *t*-BuOK (0.1 mmol) was added. TBAF = tetrabutylammonium fluoride, TBAT = tetrabutylammonium triphenyldifluorosilicate.

With the optimized reaction conditions in hand (Table 1, entry 13), the substrate scope of this copper-mediated trifluoromethylation of diaryliodonium salts with HCF_2OTf in the presence of TBAT was investigated. As shown in Scheme 2, a variety of diaryiodonium salts **1** including [Mes–I–Ar]OTf and [Mes–I–Ar]BF_4 were conveniently converted into the corresponding trifluoromethylated products **2** in moderate yields. In all cases, the products **2** arose from the reaction of less sterically hindered aryl group, which was consistent with reported Cu-catalyzed reactions of diaryliodonium salts with other nucleophiles [11,17]. The substitution of both electron-donating and electron-withdrawing groups, such as alkyl, aryl, alkenyl, ether, ester, ketone, cyano, and trifluoromethyl, on the aromatic ring did not affect the efficiency of this transformation (**2a**-**l** and **2s**). Notably, chloro or iodo containing substrates were compatible with the reaction, enabling further transformations via transition-metal-catalyzed cross-coupling reactions (**2m**-**o**). Naphthyl-type substrates (**1p** and **q**) furnished the corresponding products (**2p** and **q**) in moderate yields. A CF_3-containing tricyclic product **2r** was also obtained from **1r** under the optimized reaction conditions.

To exhibit the application value of this transformation, the trifluoromethylation of complex compounds were examined (Scheme 3). Treatment of **1t** with the standard reaction conditions gave the trifluoromethylated estrone derivative **2t** in 41% yield. Notably, this difluorocarbene-derived trifluoromethylation of compound **1u** proceeded smoothly, providing the desired product **2u**, an important precursor for the synthesis of the calcimimetic agent, Cinacalcet [18].

3. Conclusion

We have developed a convenient CuTC-mediated trifluoromethylation of diaryliodonium salts with difluoromethyltriflate in the presence of TBAT. The mild conditions and short reaction time are advantages of this process. Further studies to apply this method for the radiosynthesis of pharmacologically relevant [^{18}F]trifluoromethylarenes and heteroarenes are on progress in our laboratory.

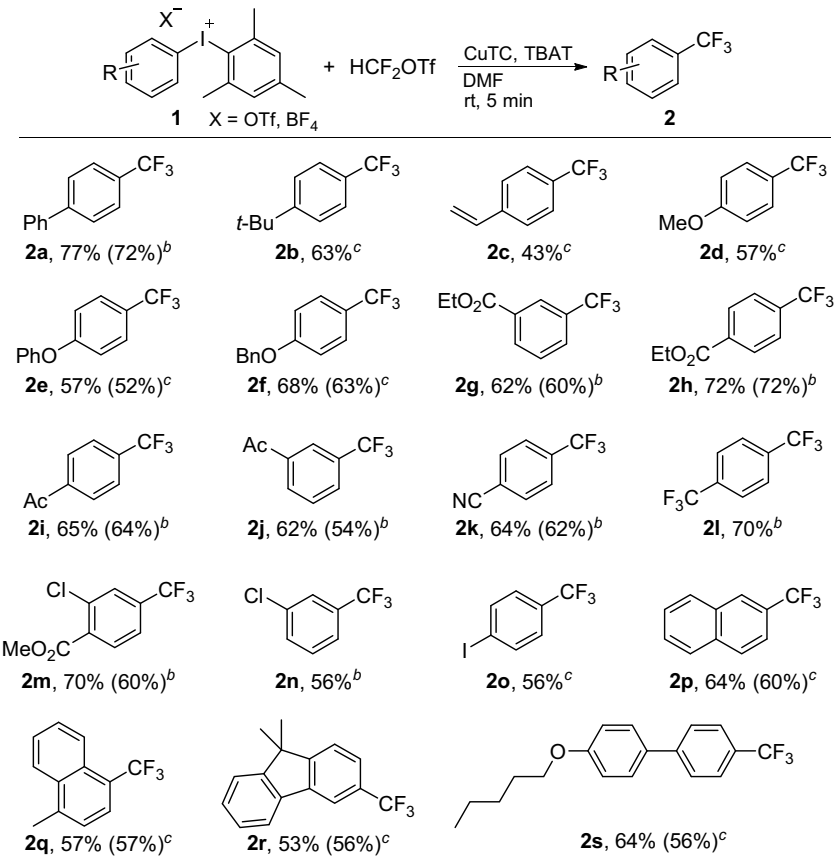
4. Experimental

4.1. General information

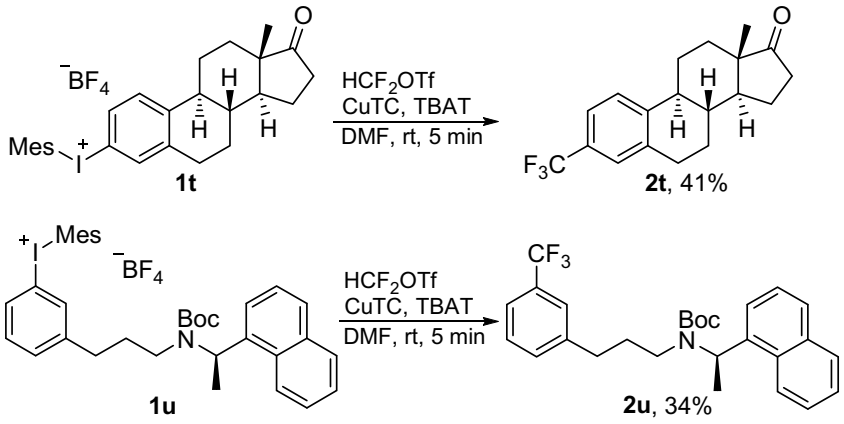
^1H NMR (TMS as the internal standard) and ^{19}F NMR spectra ($CFCl_3$ as the outside standard and low field is positive) were recorded on a Bruker AM300 or Bruker AM400 spectrometer. ^{13}C NMR was recorded on a Bruker AM400 spectrometer. Chemical shifts (δ) are reported in ppm, and coupling constants (*J*) are in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. HRMS using EI were obtained on a GC-TOF mass spectrometer. Reactions were performed under an atmosphere of nitrogen using glassware that was flame-dried under vacuum. The diaryliodonium salts were prepared from the corresponding iodoarene or arylboronic acid following the procedures below and were directly used [11,19]. All starting materials and reagents were purchased from commercial sources and used without further purification.

4.2. General procedure for the synthesis of diaryliodonium salts

Procedure A: *Preparation of diaryliodonium triflates.* The indicated iodoarene (9.0 mmol), *m*CPBA (1.20 g, 10.0 mmol), CH_2Cl_2



Scheme 2. Scope of substrates. [a]Reaction conditions: **1** (0.5 mmol), CuTc (0.5 mmol), HCF_2OTf (2.0 mmol), TBAT (1.5 mmol), DMF (10.0 mL), under N_2, rt, 5 min. Yields were determined by ^{19}F NMR spectroscopy using trifluoromethoxylbenzene as an internal standard. Yields in parentheses were isolated yields. [b]Products were obtained from the corresponding triflates. [c]Products were obtained from the corresponding tetrafluoroborates.



Scheme 3. Difluorocarbene-derived trifluoromethylation of complex compounds.

(40 mL) and mesitylene (1.39 mL, 10.0 mmol) was added to a 100 mL round-bottom flask equipped with a stir bar. The solution was cooled to 0 °C. TfOH (1.33 mL, 1.6 eq) was added dropwise over 3 min, then the reaction was allowed to warm to room temperature. After stirring for 2 h, the solvent was removed *in vacuo* and Et_2O (20 mL) was added to the mixture. The mixture was cooled to −20 °C for at least 30 min. The diaryliodonium triflates was filtered, washed with Et_2O and dried under vacuum.

Procedure B: *Preparation of diaryliodonium tetrafluoroborates.* To a 250 mL round-bottom flask equipped with a stir bar was added the indicated arylboronic acid (3.5 mmol) and CH_2Cl_2 (30 mL). The mixture was cooled to 0 °C. $BF_3{\cdot}OEt_2$ (0.48 mL, 3.9 mmol) was

added and the mixture was stirred for 10 min. Then a solution of 2-(diacetoxyiodo)mesitylene (1.42 g, 3.9 mmol) in CH_2Cl_2 (15 mL) was added dropwise to the mixture over 2 min. The reaction was allowed to warm to room temperature and stirred for 2 h. Then saturated aqueous $NaBF_4$ (70 mL) was added with vigorous stirring. After stirring for 45 min, the aqueous layer was extracted with CH_2Cl_2 (3 × 40 mL). The combined organic layers were dried over Na_2SO_4. The solvent was removed *in vacuo*, then Et_2O (20 mL) was added and the mixture was cooled to −20 °C for at least 30 min. The diaryliodonium tetrafluoroborates was filtered, washed with Et_2O and dried under vacuum.

4.3. General procedure for trifluoromethylation of diaryliodonium salts with difluoromethyltriflate for characterization by ^{19}NMR spectroscopy

An over-dried 50 mL Schlenk tube equipped with a magnetic stir bar was charged with CuTC (95.3 mg, 0.5 mmol, 1.0 eq) and TBAT (809.8 mg, 1.5 mmol, 3.0 eq). The seal tube was evacuated and backfilled with N_2. Then HCF_2OTf (233 μL, 2.0 mmol, 4.0 eq) and DMF (10.0 mL) were added by syringes. The mixture was stirred at room temperature for 5 min and diaryliodonium salt (0.5 mmol, 1.0 eq) was added under N_2. After stirring for 5 min, the reaction was quenched by H_2O and the aqueous layer was extracted with Et_2O. To the combined organic layers, trifluoromethoxybenzene (66 μL, 0.5 mmol, 1.0 eq) was then added as an internal standard. Then, the reaction mixture was directly characterized by ^{19}F NMR spectroscopy.

4.3.1. 1-(Tert-*butyl*)-4-*(trifluoromethyl)benzene* (***2b***)

Compound **2b** was prepared in 63% ^{19}F NMR yield from (4-(*tert*-butyl)phenyl)(mesityl)iodonium tetrafluoroborate (233.1 mg, 0.5 mmol) prepared by **procedure B**. ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.12 (s, 3F). This assignment matched with those previously reported [4h,11c].

4.3.2. 1-(Trifluoromethyl)-4-vinylbenzene (***2c***)

Compound **2c** was prepared in 43% ^{19}F NMR yield from mesityl (4-vinylphenyl)iodonium tetrafluoroborate (218.0 mg, 0.5 mmol) prepared by **procedure B**. ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.30 (s, 3F). This assignment matched with those previously reported [5l,11c].

4.3.3. 1-methoxy-4-(trifluoromethyl)benzene (***2d***)

Compound **2d** was prepared in 57% ^{19}F NMR yield from mesityl (4-methoxyphenyl)iodonium tetrafluoroborate (220.0 mg, 0.5 mmol) prepared by **procedure B**. ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −61.18 (s, 3F). This assignment matched with those previously reported [4h,5j].

4.3.4. 1,4-bis(Trifluoromethyl)benzene (***2l***)

Compound **2l** was prepared in 70% ^{19}F NMR yield from mesityl (4-(trifluoromethyl)phenyl)iodonium trifluoromethanesulfonate (270.1 mg, 0.5 mmol) prepared by **procedure A**. ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −63.00 (s, 3F). This assignment matched with those previously reported [6e,11c].

4.3.5. 1-Chloro-3-(trifluoromethyl)benzene (***2n***)

Compound **2n** was prepared in 56% ^{19}F NMR yield from (3-chlorophenyl)(mesityl)iodonium trifluoromethanesulfonate (253.4 mg, 0.5 mmol) prepared by **procedure A**. ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.66 (s, 3F). This assignment matched with that previously reported [20].

4.3.6. 1-iodo-4-(trifluoromethyl)benzene (***2o***)

Compound **2o** was prepared in 56% ^{19}F NMR yield from (4-iodophenyl)(mesityl)iodonium tetrafluoroborate (268.0 mg, 0.5 mmol) prepared by **procedure B**. ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.76 (s, 3F). This assignment matched with those previously reported [5k,6e].

4.4. General procedure for the preparation of trifluoromethylated arenes from trifluoromethylation of diaryliodonium salts with difluoromethyltriflate

An over-dried 50 mL Schlenk tube equipped with a magnetic stir bar was charged with CuTC (95.3 mg, 0.5 mmol, 1.0 eq) and TBAT (809.8 mg, 1.5 mmol, 3.0 eq). The seal tube was evacuated and backfilled with N_2. Then HCF_2OTf (233 μL, 2.0 mmol, 4.0 eq) and DMF (10.0 mL) were added by syringes. The mixture was stirred at room temperature for 5 min and diaryliodonium salt (0.5 mmol, 1.0 eq) was added under N_2. After stirring for 5 min, the reaction was quenched by H_2O and the aqueous layer was extracted with Et_2O. The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated under reduced vacuum. The resulting residue was purified by column chromatography or HPLC to provide the desired product.

4.4.1. 4-(Trifluoromethyl)-1,1′-biphenyl (***2a***)

Compound **2a** was prepared following the general procedure in 72% yield, starting from [1,1′-biphenyl]-4-yl(mesityl)iodonium trifluoromethanesulfonate (274.2 mg, 0.5 mmol) prepared by **procedure A**. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 7.74–7.68 (m, 4H), 7.64–7.61 (m, 2H), 7.53–7.43 (m, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.38 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 144.9, 139.9, 129.3 (q, J = 33.0 Hz), 129.1, 128.3, 127.6, 127.4, 125.8 (q, J = 3.7 Hz), 124.4 (q, J = 274.5 Hz). MS (EI): *m/z* (%) 222 (100). HRMS: Calculated for $C_{13}H_9F_3$: 222.0656; Found $[M]^+$, 222.0654.

4.4.2. 1-Phenoxy-4-(trifluoromethyl)benzene (***2e***)

Compound **2e** was prepared following the general procedure in 52% yield, starting from mesityl(4-phenoxyphenyl)iodonium tetrafluoroborate (251.1 mg, 0.5 mmol) prepared by **procedure B**. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 7.58 (d, J = 8.4 Hz, 2H), 7.40 (t, J = 7.9 Hz, 2H), 7.20 (t, J = 7.5 Hz, 1H), 7.08-7.04 (m, 4H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −61.75 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 160.7 (d, J = 1.5 Hz), 155.9, 130.3, 127.3 (q, J = 3.8 Hz), 125.0 (q, J = 33.5 Hz), 124.7, 124.4 (q, J = 272.0 Hz), 120.1, 118.0. MS (EI): *m/z* 238 (M^+). HRMS: Calculated for $C_{13}H_9F_3O$ (M^+): 238.0605; Found $[M]^+$, 238.0601.

4.4.3. 1-(Benzyloxy)-4-(trifluoromethyl)benzene (***2f***)

Compound **2f** was prepared following the general procedure in 63% yield, starting from (4-(benzyloxy)phenyl)-(mesityl)iodonium tetrafluoroborate (258.1 mg, 0.5 mmol) prepared by **procedure B**. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 7.56 (d, J = 8.7 Hz, 2H), 7.46-7.36 (m, 5H), 7.05 (d, J = 8.7 Hz, 2H), 5.12 (s, 2H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −61.47 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 161.3 (d, J = 1.6 Hz), 136.3, 128.9, 128.4, 127.6, 127.1 (q, J = 3.7 Hz), 124.6 (q, J = 272.6 Hz), 123.2 (q, J = 32.9 Hz), 115.0, 70.3. MS (EI): *m/z* 252 (M^+). HRMS: Calculated for $C_{14}H_{11}F_3O$ (M^+): 252.0755; Found, 252.0754.

4.4.4. Ethyl 3-(trifluoromethyl)benzoate (***2g***)

Compound **2g** was prepared following the general procedure in 60% yield, starting from (3-(ethoxycarbonyl)phenyl)(mesityl)iodonium trifluoromethanesulfonate (272.2 mg, 0.5 mmol) prepared by **procedure A**. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 8.28 (s, 1H), 8.21 (d, J = 7.6 Hz, 1H), 7.77 (dt, J = 7.6 Hz, J = 1.2 Hz, 1H), 7.57-7.53 (m, 1H), 4.40 (q, J = 7.2 Hz, 2H), 1.40 (t, J = 7.2 Hz, 3H). ^{19}F NMR (376 MHz,

$CDCl_3$): δ ppm −62.92 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 165.4, 132.9, 131.5, 131.1 (q, J = 32.9 Hz), 129.4 (q, J = 3.8 Hz), 129.1, 126.6 (q, J = 4.0 Hz), 123.8 (q, J = 272.6 Hz), 61.6, 14.3. MS (EI): m/z 218 (M^+). HRMS: Calculated for $C_{13}H_7F_3O$ (M^+): 218.0555; Found $[M]^+$, 218.0558.

*4.4.5. Ethyl 4-(trifluoromethyl)benzoate (**2h**)*

Compound **2h** was prepared following the general procedure in 72% yield, starting from [4-(ethoxycarbonyl)phenyl](mesityl)iodonium trifluoromethanesulfonate (272.2 mg, 0.5 mmol) prepared by **procedure A**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 8.15 (d, J = 7.8 Hz, 2H), 7.69 (d, J = 7.8 Hz, 2H), 4.41 (q, J = 7.1 Hz, 2H), 1.41 (t, J = 7.1 Hz, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −63.16 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 165.5, 134.4 (q, J = 32.6 Hz), 133.8, 130.1, 125.5 (q, J = 3.8 Hz), 123.8 (q, J = 273.3 Hz), 61.7, 14.4. MS (EI): m/z 218 (M^+). HRMS: Calculated for $C_{10}H_9F_3O_2$ (M^+): 218.0555; Found, 218.0550.

*4.4.6. 1-[4-(Trifluoromethyl)phenyl]ethanone (**2i**)*

Compound **2i** was prepared following the general procedure in 64% yield, starting from (4-acetylphenyl)(mesityl)iodonium trifluoromethanesulfonate (257.2 mg, 0.5 mmol) prepared by **procedure A**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 7.83 (d, J = 8.7 Hz, 2H), 7.66 (d, J = 8.7 Hz, 2H), 2.57 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −63.14 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 197.0, 139.7, 134.4 (q, J = 32.7 Hz), 128.6, 125.7 (q, J = 3.9 Hz), 123.6 (q, J = 272.7 Hz), 26.7. MS (EI): m/z 188 (M^+). HRMS: Calculated for $C_9H_7F_3O$ (M^+): 188.0449; Found, 188.0447.

*4.4.7. 1-[3-(Trifluoromethyl)phenyl]ethanone (**2j**)*

Compound **2j** was prepared following the general procedure in 54% yield, starting from (3-acetylphenyl)(mesityl)iodonium trifluoromethanesulfonate (257.2 mg, 0.5 mmol) prepared by **procedure A**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 8.16 (s, 1H), 8.09 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.57 (t, J = 7.8 Hz, 1H), 2.60 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.95 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 196.1, 137.7, 131.5, 131.3 (q, J = 33.0 Hz), 129.5 (q, J = 3.6 Hz), 129.4, 125.1 (q, J = 3.2 Hz), 123.8 (q, J = 273.0 Hz), 26.5. MS (EI): m/z 188 (M^+). HRMS: Calculated for $C_{13}H_7F_3O$ (M^+): 188.0449; Found, 188.0448.

*4.4.8. 4-(Trifluoromethyl)benzonitrile (**2k**)*

Compound **2k** was prepared following the general procedure in 62% yield, starting from (4-cyanophenyl)(mesityl)iodonium trifluoromethanesulfonate (248.6 mg, 0.5 mmol) prepared by **procedure A**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 7.83 (d, J = 6.3 Hz, 2H), 7.76 (d, J = 6.3 Hz, 2H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −63.55 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 134.7 (q, J = 33.5 Hz), 132.8, 126.3 (q, J = 3.6 Hz), 123.2 (q, J = 273.1 Hz), 117.6, 116.2. MS (EI): m/z 171 (M^+). HRMS: Calculated for $C_8H_4F_3N$ (M^+): 171.0296; Found, 171.0298.

*4.4.9. 2-Chloro-4-(trifluoromethyl)phenyl acetate (**2m**)*

Compound **2m** was prepared following the general procedure in 60% yield, starting from (3-chloro-4-(methoxycarbonyl)phenyl)(mesityl)iodonium trifluoromethanesulfonate (282.4 mg, 0.5 mmol) prepared by **procedure A**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 8.11 (d, J = 1.6 Hz, 1H), 7.67 (dd, J = 8.4 Hz, J = 1.4 Hz, 1H), 7.59 (d, J = 8.0 Hz, 1H), 3.97 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.89 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 164.9, 137.9, 132.0, 130.8, 129.5 (q, J = 33.8 Hz), 129.2 (q, J = 3.6 Hz), 128.7 (q, J = 3.9 Hz), 123.4 (q, J = 272.6 Hz), 52.9. MS (EI): m/z 238 (M^+). HRMS: Calculated for $C_9H_6ClF_3O_2$ (M^+): 238.0008; Found, 238.0003.

*4.4.10. 2-(Trifluoromethyl)naphthalene (**2p**)*

Compound **2p** was prepared following the general procedure in 60% yield, starting from mesityl(naphthalen-2-yl)iodonium tetrafluoroborate (230.0 mg, 0.5 mmol) prepared by **procedure B**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 8.19–8.14 (m, 1H), 8.00-7.89 (m, 3H), 7.67–7.56 (m, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.26 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 134.6, 132.2, 129.0, 128.8, 128.1, 127.9, 127.7 (q, J = 32.4 Hz), 127.2, 125.7 (q, J = 4.5 Hz), 124.4 (q, J = 272.5 Hz), 121.4 (q, J = 3.4 Hz). MS (EI): m/z 196 (M^+). HRMS: Calculated for $C_{11}H_7F_3$ (M^+): 196.0500; Found, 196.0504.

*4.4.11. 1-Methyl-4-(trifluoromethyl)naphthalene (**2q**)*

Compound **2q** was prepared following the general procedure in 57% yield, starting from mesityl(4-methylnaphthalen-1-yl)iodonium tetrafluoroborate (237.0 mg, 0.5 mmol) prepared by **procedure B**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 8.30–8.27 (m, 1H), 8.11–8.08 (m, 1H), 7.79 (d, J = 7.5 Hz, 1H), 7.67–7.63 (m, 2H), 7.34 (d, J = 7.5 Hz, 1H), 2.74 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −59.25 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 139.7, 133.2, 129.2, 127.3, 126.5, 125.2 (q, J = 273.3 Hz), 125.1, 124.9, 124.9, 124.6 (q, J = 6.0 Hz), 124.5 (q, J = 30.1 Hz), 19.8. MS (EI): m/z 210 (M^+). HRMS: Calculated for $C_{12}H_9F_3$ (M^+): 210.0656; Found, 210.0651.

*4.4.12. 9,9-Dimethyl-3-(trifluoromethyl)-9H-fluorene (**2r**)*

Compound **2r** was prepared following the general procedure in 56% yield, starting from (9,9-dimethyl-9H-fluoren-2-yl)(mesityl) iodonium tetrafluoroborate (263.1 mg, 0.5 mmol) prepared by **procedure B**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 7.82-7.73 (m, 2H), 7.68 (d, J = 0.8 Hz, 1H), 7.62 (dd, J = 7.6 Hz, J = 0.8 Hz, 1H), 7.50-7.48 (m, 1H), 7.41-7.39 (m, 2H), 1.53 (s, 6H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −61.62 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 154.3, 154.1, 142.9, 137.9, 129.3 (q, J = 32.0 Hz), 128.6, 127.4, 124.8 (q, J = 272.5 Hz), 124.5 (q, J = 3.6 Hz), 123.0, 120.9, 120.2, 119.8 (q, J = 3.8 Hz), 47.2, 27.1. MS (EI): m/z 262 (M^+). HRMS: Calculated for $C_{13}H_7F_3O$ (M^+): 262.0969; Found, 262.0974.

*4.4.13. 4-(Pentyloxy)-4′-(trifluoromethyl)-1,1′-biphenyl (**2s**)*

Compound **2s** was prepared following the general procedure in 56% yield, starting from mesityl(4′-(pentyloxy)-[1,1′-biphenyl]-4-yl)iodonium (286.1 mg, 0.5 mmol) prepared by **procedure B**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 7.68-7.63 (m, 4H), 7.52 (d, J = 9.0 Hz, 2H), 6.99 (d, J = 9.0 Hz, 2H), 4.00 (t, J = 6.4 Hz, 2H), 1.86-1.79 (m, 2H), 1.49-1.34 (m, 4H), 0.95 (t, J = 7.0 Hz, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.35 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 159.6, 144.6, 132.1, 128.8 (q, J = 32.5 Hz), 128.5, 127.0, 125.8 (q, J = 3.7 Hz), 124.6 (q, J = 271.8 Hz), 115.2, 68.3, 29.1, 28.4, 22.6, 14.2. IR (thin film) ν 2959, 2938, 2874, 1606, 150, 1326, 1275, 1169, 1124, 1072, 909, 825, 734 cm^{-1}. MS (EI): m/z 308 (M^+). HRMS: Calculated for $C_{18}H_{19}F_3O$ (M^+): 308.1388; Found, 308.1385.

*4.4.14. (8R,9S,13S,14S)-13-Methyl-3-(trifluoromethyl)-7,8,9,11,12,13,15,16-octahydro-6H-cyclopenta[a]phenanthren-17(14H)-one (**2t**)*

Compound **2t** was prepared following the general procedure in 41% yield, starting from mesityl((8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)iodonium tetrafluoroborate (293.1 mg, 0.5 mmol) prepared by **procedure B**. ^1H NMR (300 MHz, $CDCl_3$): δ ppm 7.39 (m, 2H), 7.35 (s, 1H), 2.98-2.94 (m, 2H), 2.55-2.42 (m, 2H), 2.36-2.31 (m, 1H), 2.20-1.98 (m, 4H), 1.67-1.45 (m, 6H), 0.91 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.38 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 143.9, 137.4, 128.2 (q, J = 32.2 Hz), 125.9, 125.9 (q, J = 3.8 Hz), 125.8, 124.5 (q, J = 273.3 Hz), 122.6 (q, J = 3.6 Hz), 50.6, 48.0, 44.6, 37.9, 35.9, 31.6, 29.4, 26.3, 25.7, 21.7, 13.9. MS (EI): m/z 322 (M^+). HRMS: Calculated for $C_{19}H_{21}F_3O$ (M^+): 322.1545; Found, 322.1548.

*4.4.15. (R)-*tert*-Butyl (1-(naphthalen-1-yl)ethyl)(3-(3-(trifluoromethyl)phenyl)propyl)carbamate (**2u**)*

Compound **2u** was prepared following the general procedure in 34% yield, starting from (*R*)-(3-(3-((*tert*-butoxycarbonyl)(1-(naphthalen-1-yl)ethyl)amino)propyl)phenyl)(mesityl)iodonium (360.7 mg, 0.5 mmol) prepared by **procedure B**. ^{1}H NMR (300 MHz, $CDCl_3$): δ ppm 8.18 (d, *J* = 6.0 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.55-7.47 (m, 2H), 7.43-7.33 (m, 3H), 7.24-7.20 (m, 1H), 6.91-6.83 (m, 2H), 6.21-6.04 (m, 1H), 3.11-2.75 (m, 2H), 2.16-2.14 (m, 2H), 1.61-1.50 (m, 12H), 1.27-1.21 (m, 1H), 0.78-0.67 (m, 1H). ^{19}F NMR (376 MHz, $CDCl_3$): δ ppm −62.50 (s, 3F). ^{13}C NMR (100.7 MHz, $CDCl_3$): δ ppm 155.5, 142.7, 136.8, 133.8, 132.5, 131.6, 130.5 (q, *J* = 31.9 Hz), 128.8, 128.7, 128.6, 126.6, 126.0, 125.7, 125.1, 124.9 (q, *J* = 3.7 Hz), 124.3 (q, *J* = 272.5 Hz), 124.3, 122.6, 79.8, 49.4, 42.1, 33.2, 30.9, 28.7, 17.1. MS (EI): *m*/*z* 457 (M^+). HRMS: Calculated for $C_{27}H_{30}F_3NO_2$ (M^+): 457.2229; Found, 457.2231.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jfluchem.2016.04.008.

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